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Indian Standard

CODE OF PRACTICE FOR TREATMENT OF WATER FOR MARINE BOILERS

(Second Reprint DECEMBER 1976)

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Indian Standard

CODE OF PRACTICE FOR TREATMENT OF WATER FOR MARINE BOILERS

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(*Continued on page* 2)

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AMENDMENT NO. 1 JULY 2010
TO
IS 1813 : 1961 CODE OF PRACTICE FOR TREATMENT OF WATER FOR MARINE BOILERS

(*Page* 10, *Appendix* B, *clause* **B-0.2)** — Substitute 'IS 1070 : 1992 Reagent grade water (*third revision*)' *for* 'IS 1070 : 1960 Specification for Water, Distilled Quality (*Revised*)'.

(CHD 13)

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(*Continued from page* 1)

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CODE OF PRACTICE FOR TREATMENT OF WATER FOR MARINE BOILERS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 April 1961, after the draft finalized by the Water Sectional Committee had been approved by the Chemical Division Council.

0.2 In the case of land boilers it is practicable to treat the feed water in an external water softening plant but due to the limited space available on a ship, it is not possible to do so for marine boilers except on the largest of ships. Internal treatment has, therefore, to be used for marine boilers. The steam used for prime movers is condensed and recovered for use as a boiler feed; thus a comparatively small quantity of water is required as make-up. In many cases, evaporated, that is, distilled water is used as make-up so that the feed water is always of a uniformly good character, the only impurities being those derived from leakage of condensers and carry-over from evaporators. These can be easily put right and the contamination avoided. However, many ships do not use evaporated or distilled water for make-up but use fresh water taken at ports, while some have to use, when in emergency, unevaporated sea water.

0.3 In order to make this standard comprehensive, recommended composition for boiler water for marine boilers is given in Appendix A. Also, mention has been made of the treatment necessary at the time of initial filling and for protection of standing boilers.

0.4 Detailed methods of test for water are not included in this code. These will be covered by separate Indian Standards. However, routine tests for controlling the treatment are given in Appendix B.

0.5 This code is one of a series of Indian Standard Codes of Practice on treatment of water for boilers. The other Indian Standard Code published so far in the series is *IS : 1680-1960 Code of Practice for Treatment of Water for Land Boilers.

0.6 In preparing this code, assistance has been derived from B.S. 1170 : 1957 Treatment of Water for Marine Boilers, issued by the British Standards Institution.

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*Since revised.

0.7 Wherever a reference to any Indian Standard appears in this code, it shall be taken as a reference to the latest version of the standard.

0.8 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*).

0.9 Metric system has been adopted in India and all quantities appearing in this standard have been given in this system. Non-metric values to which the industry has been accustomed are also given in brackets, wherever necessary, for the sake of smooth changeover by December 1966.

1. SCOPE

1.1 This standard lays down the methods to be adopted for treatment of water for marine boilers.

2. OBJECT OF TREATMENT

2.1 The type of treatment, and its various details, best suited to a particular case shall depend on the nature and extent of the impurities to be dealt with and on the use and efficiency of mechanical operations governing blow-down, de-aeration, oil filters, etc; but in all cases the aims are the same as with land boilers and may be summarised thus:

a) Complete removal of all scale-forming salts and oil from boiler water by precipitation in the form of non-adhering and smooth flowing sludge and maintenance of small but adequate reserve of precipitating agents;

b) Reduction of corrosion to a minimum by maintaining sufficient alkalinity in the boiler water and reduction of dissolved oxygen to the minimum; and

c) Prevention of priming and foaming under all operating conditions.

3. CHEMICALS USED IN TREATMENT OF WATER

3.1 Sodium Phosphate (for Prevention of Scale) — Sodium phosphate is used for the precipitation of calcium salts. There are three phosphates that may be used for this purpose:

a) Disodium phosphate,

b) Trisodium phosphate, and

c) Sodium metaphosphate (sodium hexametaphosphate).

In a boiler, sodium metaphosphate and disodium phosphate are converted into trisodium phosphate which remains stable even under high boiler pressures and temperatures. The choice depends upon the alkalinity requirements of the feed water. All the three phosphates produce sodium carbonate in the boiler water when they react with the carbonate hardness of the feed water. Sodium metaphosphate produces the least and trisodium phosphate the greatest alkalinity. When using a feed water with high alkaline hardness, sodium metaphosphate producing the least amount of sodium carbonate is preferable and when feed water of low alkaline hardness is used, trisodium phosphate would be more suitable. Sometimes the sodium carbonate thus produced is insufficient and additional free sodium carbonate has to be added to maintain the requisite alkalinity.

3.2 Soda Ash (for Maintaining Alkalinity) — Soda ash is used when it is necessary to supplement the alkalinity produced by sodium phosphate. Under boiler conditions, sodium carbonate is partly converted into sodium hydroxide which precipitates non-carbonate hardness due to magnesium salts as magnesium hydroxide. Instead of soda ash, caustic soda may also be used for maintaining the required alkalinity and precipitation of magnesium but due to the latter's caustic nature, soda ash is preferred.

3.3 Sodium Sulphite (for Prevention of Corrosion by Eliminating Dissolved Oxygen) — The dissolved oxygen shall be removed mechanically as efficiently as possible from the feed water before it reaches the boiler. For boilers with pressure up to 18 kg/cm^2 (or 250 lb/in^2) unless the dissolved oxygen content is uniformly maintained below 0·07 ppm (or 0·05 ml per litre), or for boilers with pressure above 18 kg/cm^2 (or 250 lb/in^2) unless the dissolved oxygen content is uniformly maintained below 0·03 ppm (or 0·02 ml per litre), sodium sulphite shall be added. It is a safe and good practice, especially with water tube boilers, to de-aerate the water and add sodium sulphite as a routine. A reserve of 20 to 30 ppm of sodium sulphite shall be maintained in the boiler water at all times.

3.4 Sodium Sulphate or Sodium Nitrate (for Prevention of Caustic Embrittlement) — In the case of riveted drums and welded drums which have not been stress-relieved, sodium sulphate shall be added in sufficient amount to maintain the ratio of sodium sulphate/caustic alkalinity (as NaOH) above 2·5 at all times. If sodium nitrate is used, the ratio sodium nitrate/total alkalinity (as NaOH) shall be above 0·4. This is the best recommendation that can be made in the light of present knowledge on the subject which is still under investigation. But this treatment does not prevent the spread of any cracks existing prior to the use of the chemical.

3.5 Coagulants — In the internal process of water softening by addition of chemicals, the impurities are precipitated and the precipitate remains in the boiler. It is of highest importance that the precipitate should be non-adherent in character. For this purpose, some organic substances, such as tannin, starch, etc, or sodium aluminate shall be added along with other chemicals. These convert the precipitate into a smooth, free flowing, non-adhering sludge which can be readily removed with blow-down water.

3.6 De-Oiling Agents — Oil shall be removed by any of the usual mechanical methods used. The traces of oil remaining in the water are generally trapped by starch and the phosphate precipitate.

4. METHODS OF TREATMENT

4.0 Three types of treatment which are intended for various types of make-up feed are prescribed in **4.1** to **4.3**.

4.1 Make-Up Consisting of Evaporated Water or Unevaporated Soft Water

4.1.1 *Treatment by Standard Mixture* — When the boilers are fed with condensate with make-up of evaporated water, the character of feed water remains very nearly the same all the time. The only impurities found are those obtained from leakage of condensers or carry-over from the evaporators. These impurities are very small in amount and of uniform character. The proportion of chemicals to be added thus remains the same and only the quantity has to be varied as required to obtain the necessary results. The condition remains almost the same if instead of using evaporated water as make-up, fresh water of low hardness is used direct. Under these conditions, adequate treatment can be effected by the addition of a standard mixture of the following composition:

Soda ash	39 percent
Anhydrous disodium phosphate	48 percent
Starch	13 percent

In addition to this, sodium sulphite and sodium sulphate shall be added for prevention of corrosion and caustic embrittlement respectively. The boiler water shall be tested from time to time for hardness and alkalinity and the mixture or the other chemicals added to maintain the recommended conditions as given in Appendix A. This mixture gives quite satisfactory results and chemical boiler mixtures of approximately this composition are available at ports. This treatment will not only prevent formation of scale but will remove all previously formed scale. Care should, therefore, be taken when using this method in boilers with previous scale in existence.

4.1.1.1 *Adjustment of dose* — Test the boiler water sample for hardness and alkalinity to phenolphthalein. If the phenolphthalein alkalinity is lower than the lower limit shown in Appendix A and residual hardness is present, increase the amount of mixture next time at the rate of 10 percent and continue adding such amount till the desired result is obtained. If the alkalinity is too high and hardness is zero, no mixture shall be added at all till the alkalinity drops down to the desired figure.

4.1.1.2 *Limits of treatment* — Normally this treatment shall be followed and the condition of boiler water maintained within limits shown in Appendix A. However, when there is gross contamination due to heavy leakage of condensers of serious carry-over from evaporators, a very large quantity of the mixture would be required to maintain these limits. This will also produce an excessive amount of precipitate as well as dissolved solids both of which are undesirable and harmful. Under these circumstances, the treatment shall be stopped.

4.1.2 *Treatment by Separate Chemicals* — If it is desired to use separate chemicals instead of the standard mixture mentioned in **4.1.1**, the boiler water shall be tested for phosphate and alkalinity.

4.1.2.1 If phosphate is less than 30 ppm (as PO_4) in the boiler water, sodium metaphosphate shall be added. If phosphate is more than 70 ppm in the boiler water, no phosphate shall be added till the concentration drops down to below 70 ppm. When phosphate is to be added, sodium metaphosphate at the rate of 14 g/metric tonne (or 0·5 oz/ton) for every 10 ppm phosphate deficiency in the boiler water shall be added. In the absence of sodium metaphosphate, equivalent amounts of other sodium phosphates shall be added.

4.1.2.2 If the phenolphthalein alkalinity is lower than what is desired, increase soda ash by 55 g/metric tonne (or 2 oz/ton) of boiler water for every 50 ppm of alkalinity deficiency. If phenolphthalein alkalinity is too high, stop adding soda ash till the condition falls down to the desired point.

4.1.2.3 *Limits of treatment* — These shall be as given in Appendix A.

4.2 Make-Up Consisting of Unevaporated Hard Water

4.2.1 The chemicals to be used are the same as those used in the treatment of water described in **4.1**, namely sodium phosphate, soda ash, some coagulant, sodium sulphite and sodium sulphate. As the character of water taken at different ports may be different, it would not be practicable to prepare stock mixtures for treatment of such water. But if the water taken from ports happens to be very similar, mixtures to suit them could be prepared by specialists who supply them. The usual course is to test the boiler water for phosphate and alkalinity and add sodium metaphosphate and soda ash as explained in **4.1.2**. It is preferable to use sodium metaphosphate as in case disodium or trisodium phosphate were used a large amount of alkali would be produced. In some cases, these latter phosphates also may be used satisfactorily.

4.2.1.1 *Limits of treatment* — Boiler water should be maintained within limits given in Appendix A.

4.3 Make-Up Consisting of Unevaporated Sea Water or Water Heavily Contaminated with Sea Water — In this case, it is not practicable to precipitate all the hardness with phosphates as it would be too expensive and the concentration of dissolved solids and suspended solids in the boiler water would also be excessive and give rise to priming and foaming. The only treatment to be applied in this case would be to prevent corrosion and prevent priming and foaming. The water should be maintained alkaline to phenolphthalein. No definite limits of alkalinity can be recommended. The boiler water should show a pink colour when a few drops of phenolphthalein solution are added to it. The density of boiler water should be maintained within 4/32 on a salinometer, that is, approximately 125 000 ppm. The above treatment will afford a certain amount of protection against corrosion while addition of suitable antifoams will minimize foaming and priming.

5. CARE OF BOILERS

5.1 Initial Filling — Boilers shall be initially filled with evaporated water as far as practicable. If this water is not available, the usual feed water may be used. Standard boiler mixture shall be added at the rate of 0·5 kg/metric tonne (or 1 lb/ton) of water and 0·1 kg of sodium sulphite per metric tonne (or 4 oz/ton) of water shall also be used. Sodium sulphate shall be added to maintain the desired ratio in the case of riveted boiler drums and non-stress relieved drums.

5.2 Protection of Standing Boilers

5.2.1 *Short Periods* — When boilers are to be kept at rest for short periods, they shall be filled completely with water having 0·25 kg of soda ash and 0·1 kg of anhydrous sodium sulphite per metric tonne (or 0·5 lb soda ash and 4 oz anhydrous sodium sulphite per ton) of water. Boilers thus filled may be started without change of water but an appropriate quantity of phosphate shall be added to precipitate calcium salts.

5.2.2 *Long Periods* — When boilers are to be kept idle for longer periods, they shall be emptied, thoroughly dried, and deposits removed. Trays of quick lime shall be placed in the drums and the boilers sealed.

APPENDIX A

(*Clauses* 0.3, 4.1.1, 4.1.1.1, 4.1.1.2, 4.1.2.3 *and* 4.2.1.1)

RECOMMENDED COMPOSITION OF BOILER WATER FOR MARINE BOILERS

Sl No.	Characteristic	Recommended Limits for		
		Water Tube Boilers	Scotch Marine Boilers	Vertical Boilers
(1)	(2)	(3)	(4)	(5)
i)	Hardness (as $CaCO_3$)	Nil	Nil	Nil
ii)	Alkalinity to phenolphthalein (as $CaCO_3$), parts per million	150 to 350	300 to 700	200 to 500
iii)	Chlorides (as Cl), parts per million	50 to 350	2 000 (3 600)* *Max*	950 (1 500)* *Max*
iv)	Dissolved solids, parts per million	500 to 2 000	7 000(15 000)* *Max*	4 200(6 000)* *Max*
v)	Phosphates (as PO_4), parts per million	30 to 70	30 to 70	30 to 70
vi)	Sodium sulphite (as Na_2SO_3), parts per million	20 to 30	20 to 30	20 to 30
vii)	†Ratio of sodium sulphate/caustic alkalinity (as NaOH) *Or*	Above 2·5	Above 2·5	Above 2·5
	†Ratio of sodium nitrate/total alkalinity (as NaOH)	Above 0·4	Above 0·4	Above 0·4

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*The figures in parentheses are maximum values which can be carried into the boilers in the event of sea water leakage or evaporator carry-over, above which priming and carry-over is likely to occur.

†For riveted and non-stress relieved boilers only.

APPENDIX B

(*Clause* 0.4)

CONTROL TESTS FOR WATER FOR MARINE BOILERS

B.0. GENERAL

B-0.1 Only such methods as are required for routine tests for controlling the quality of boiler water for marine boilers have been described in this appendix.

B-0.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water [*see* IS : 1070-1960 Specification for Water, Distilled Quality (*Revised*)] shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals which do not contain impurities that affect the results of analysis.

B-1. ALKALINITY TO PHENOLPHTHALEIN AND CAUSTIC ALKALINITY

B-1.1 Reagents

B-1.1.1 *Phenolphthalein Indicator Solution* — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit [conforming to IS : 323-1959 Specification for Rectified Spirit (*Revised*)].

B-1.1.2 *Standard Sulphuric Acid* — 0·02 N.

B-1.1.3 *Barium Chloride Solution* — 10 percent (*w/v*).

B-1.2 Procedure

B-1.2.1 *For Alkalinity to Phenolphthalein* — Measure out 100 ml of the sample (filtered, if cloudy) by means of a graduated cylinder and transfer to a casserole or porcelain basin. Add 10 drops of phenolphthalein indicator solution. If the water turns pink on the addition of the indicator, run in standard sulphuric acid slowly from the burette, at the same time stirring with a glass rod, until the colour just disappears. Record the number of millilitres of acid (p) used.

B-1.2.2 *For Caustic Alkalinity* — Take 100 ml of fresh sample (filtered, if cloudy) in a casserole or porcelain dish. Add 10 ml of barium chloride solution and 10 drops of phenolphthalein indicator solution. Run in standard sulphuric acid slowly from the burette, at the same time stirring with a glass rod, until the colour just disappears. Record the number of millilitres of acid (pb) used.

B-1.3 Calculation

a) Alkalinity to phenolphthalein (as $CaCO_3$), parts per million $= 10\,p$

b) Caustic alkalinity (as $CaCO_3$), parts per million $= 10\,pb$

NOTE — Where high alkalinity is expected, smaller quantity of the sample may be used.

B-2. CHLORIDES

B-2.1 Reagents

B-2.1.1 *Methyl Orange Indicator* — Dissolve 0·1 g of methyl orange in 100 ml of water.

B-2.1.2 *Standard Sulphuric Acid* — Same as in **B-1.1.2.**

B-2.1.3 *Potassium Chromate Indicator Solution* — Dissolve 5 g of potassium chromate in 100 ml of water.

B-2.1.4 *Standard Silver Nitrate Solution* — N/35·5.

B-2.2 Procedure — Measure out 100 ml of the filtered sample by means of a graduated cylinder and transfer to a casserole. Add three drops of methyl orange indicator. Run in standard sulphuric acid slowly from the burette, at the same time stirring with a glass rod until the colour just changes to orange. To the neutralized solution, add 10 drops of potassium chromate indicator solution. Add standard silver nitrate solution from a burette, stirring well during the addition until the yellow colour just changes to reddish brown tinge. Record the number of millilitres (v) of standard silver nitrate solution used.

B-2.3 Calculation

Chlorides (as Cl), parts per million $= 10\,v$

NOTE — Where a high chloride content is expected, a smaller volume of the sample should be taken and diluted to 100 ml for titration.

B-3. TOTAL HARDNESS

B-3.3 Two alternative methods, the Soap Titration Method and the EDTA Method are prescribed for the determination of total hardness.

B-3.1 Soap Titration Method

B-3.1.1 *Reagents*

B-3.1.1.1 *Phenolphthalein indicator solution* — same as in **B-1.1.1.**

B-3.1.1.2 *Dilute sodium hydroxide solution* — approximately 0·1 N.

B-3.1.1.3 *Standard Wanklyn's soap solution*

a) *Preparation of Wanklyn's solution* — Dissolve 8·0 g of re-distilled oleic acid in a mixture of 60 ml of denatured spirit [conforming to IS : 324-1959 Specification for Ordinary Denatured Spirit (*Revised*)] and 40 ml of water. Add a few drops of phenolphthalein indicator and neutralize with an alcoholic solution of potassium hydroxide (about 75 ml of an approximately 2 percent solution will be required). Dilute to about 900 ml with alcohol (prepared by diluting 60 volumes of denatured spirit with 40 volumes of water).

b) *Standardization of Wanklyn's solution*

  1) *Preparation of standard calcium chloride solution* — Dissolve 1·000 g of calcium carbonate in a little dilute hydrochloric acid in a covered beaker. Evaporate to dryness (avoiding excessive heating), take up the residue in a little water, wash the cover glass allowing the washings to run into the beaker, evaporate to dryness again and then repeat the process. Dissolve the final residue in water, transfer to a 1 000-ml measuring flask, wash the beaker adding the washings to the flask, and make up to 1 000 ml with water. One millilitre of the solution is equivalent to 1·0 mg of calcium carbonate ($CaCO_3$).

  2) *Standardization of the soap solution* — By means of a pipette, introduce 10 ml of standard calcium chloride solution into a 250-ml glass-stoppered cylindrical bottle. Dilute with 90 ml of freshly boiled and cooled water. Add the soap solution a little at a time from a burette, shaking vigorously after each addition until a lather is obtained which remains for 5 minutes when the bottle is laid on its side, and note the burette reading. Repeat the determination and take a mean (m) of three readings. Make a blank (lather correction) determination on 100 ml of freshly boiled and cooled water. Repeat the determination and take a mean (c) of three readings.

  $$\text{Strength of soap solution (factor)} = \frac{10}{m-c}$$

c) *Adjustment of strength of soap solution to one millilitre$\equiv$one milligram of calcium carbonate* ($CaCO_3$) — From the factor obtained, subtract 1 and multiply the remainder by 100 and let the figure obtained be y. To each 100 ml of the original strong solution, add y ml of alcohol (prepared by diluting 60 volumes of denatured spirit with 40 volumes of water). Mix thoroughly and check the strength.

B-3.1.2 *Procedure* — Take 100 ml of sample in a 250-ml glass-stoppered bottle. Add a few drops of phenolphthalein indicator. If a pink colour is not produced, add dilute sodium hyrdoxide solution drop by drop till a faint pink colour is produced. Now add standard Wanklyn's solution from a burette 0·5 ml at a time, shaking vigorously until a lather begins to form. When this is observed, reduce the quantity of soap solution to 0·2 ml each time till a lather is formed which remains unbroken for five minutes when the bottle is placed on its side.

B-3.1.3 *Calculation*

$$\text{Total hardness (as } CaCO_3\text{), parts per million} = 10\,(V - c)$$

where

V = volume in ml of soap solution required with 100 ml of the sample, and

c = volume in ml of soap solution required with 100 ml of water [*see* **B-3.1.1.3** (b) (2)]

B-3.2 EDTA Method

B-3.2.1 *Reagents*

B-3.2.1.1 *Buffer solution* 1 — Dissolve 40 g of borax ($Na_2B_4O_7.10H_2O$) in approximately 800 ml of water. Dissolve 10 g of sodium hydroxide and 5 g of sodium sulphide in 100 ml of water. When cool, mix the two solutions and dilute to 1 000 ml with water.

B-3.2.1.2 *Indicator solution* 1 — To 30 ml of water, add 0·5 ml of 1 N sodium carbonate solution. Add 0·5 g of eriochrome black T (solochrome black T) and mix. Make up to 100 ml with isopropanol.

B-3.2.1.3 *Standard calcium chloride solution*— same as in **B-3.1.1.3** (b) (1).

B-3.2.1.4 *EDTA solution* — Dissolve 4·0 g of disodium ethylenediamine tetra-acetate dihydrate in approximately 800 ml of water. Add 0·86 g of sodium hydroxide (21·5 ml of 1 N solution) and 0·1 g of magnesium chloride ($MgCl_2.6H_2O$). Titrate against standard calcium chloride solution (10 ml diluted to 50 ml) as described in **B-3.2.2** and adjust so that one millilitre is equivalent to one milligram of calcium carbonate.

B-3.2.1.5 *Buffer solution* 2 — Dissolve 67·5 g of ammonium chloride in 570 ml of ammonium hydroxide (sp gr 0·90) and dilute to 950 ml with water. Dissolve 0·616 g of magnesium sulphate ($MgSO_4.7H_2O$) and 0·93 g of disodium ethylenediamine tetra-acetate dihydrate in 50 ml of water and add to the ammonium hydroxide-chloride mixture.

B-3.2.1.6 *Indicator solution* 2 — Dissolve 0·5 g of eriochrome black T and 4·5 g of hydroxylamine hydrochloride in 100 ml of rectified spirit [conforming to IS : 323-1959 Specification for Rectified Spirit (*Revised*)].

B-3.2.2 *Procedure* — Measure 50 ml of the sample into a 250-ml Erlenmeyer flask. Add 0·5 ml of buffer solution 1 and mix ; add 4 to 6 drops of indicator solution 1 and mix. Titrate immediately with EDTA solution with continuous mixing until the colour changes from red to blue. As the end point is approached, the solution shows some blue coloration but a definite reddish tinge can still be observed and is discharged at the end point. The duration of the titration should not exceed 5 minutes, measured from the time of the addition of the buffer solution. With exceptionally hard water, it is advisable to add the indicator solution 1 before the addition of the buffer solution 1.

B-3.2.2.1 It is recommended that for hardness below 100 ppm, 100 ml of sample be used with 1·0 ml of the buffer solution 1 and 7 to 9 drops of indicator solution 1, whereas for hardness appreciably above 200 ppm, a smaller volume than 50 ml of sample be taken and diluted to 50 ml with water. For waters of very low hardness, softened by the base-exchange process, take 500 ml of the sample. Add 2 ml of the buffer solution 2 and 6 drops of the indicator solution 2.

NOTE — To avoid over-titrating, it is suggested that 40 to 45 ml of the sample be titrated approximately to the end point, and then the remainder of the sample added and the titration continued carefully to the final end point.

B-3.2.3 *Calculation* — Calculate as follows and express the result to the nearest 5 ppm:

$$\text{Total hardness (as CaCO}_3\text{), parts per million} = \frac{1\,000\,V_1}{v}$$

where

V_1 = volume in ml of the EDTA solution used in titration, and
v = volume in ml of the sample taken for the test.

B-4. PHOSPHATES

B-4.1 Reagents

B-4.1.1 *Concentrated Nitric Acid* — conforming to *IS : 264-1950 Specification for Nitric Acid.

B-4.1.2 *Dilute Sulphuric Acid* — 1 : 4 (*v/v*).

B-4.1.3 *Phosphate Reagent A* — Dissolve 5 g of ammonium molybdate in 100 ml of 1 N sulphuric acid.

B-4.1.4 *Phosphate Reagent B* — Dissolve 0·2 g of *p*-methylaminophenol sulphate (metol or photol) in 100 ml of water and add 20 g of sodium bisulphite. Keep in well-filled tightly-stoppered bottle. The reagent shall not be kept for more than two weeks.

B-4.1.5 *Standard Phosphate Solution* — Dissolve 0·358 g of dry potassium dihydrogen phosphate (KH_2PO_4) in one litre of water. This stock solution shall be kept in the dark. Dilute 10 ml of the stock solution to one litre. One millilitre of the diluted solution is equivalent to 0·002 5 mg of phosphate (as PO_4).

B-4.2 Procedure — Evaporate 50 ml of the sample with 3 ml of concentrated nitric acid to dryness on a steam-bath. Warm the residue with one millilitre of dilute sulphuric acid, add 20 ml of water and one millilitre each of phosphate reagents *A* and *B*. Heat at 60°C for 5 minutes. Match the blue colour produced against a series of standard phosphate solutions treated in the same manner.

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* Since revised.

B-4.3 Calculation

$$\text{Phosphates (as } PO_4 \text{), parts per million} = \frac{2{\cdot}5\, v_1}{v}$$

where

v_1 = volume in ml of standard phosphate solution which matches the colour produced with the sample, and

v = volume in ml of the sample taken for the test.

B-5. SODIUM SULPHITE

B-5.1 Reagents

B-5.1.1 *Dilute Hydrochloric Acid* — Add 200 ml of concentrated hydrochloric acid (conforming to *IS : 265-1950 Specification for Hydrochloric Acid) to 100 ml of water.

B-5.1.2 *Standard Iodate-Iodide Solution* — Dissolve 0·710 g of potassium iodate in about 160 ml of water. Add 7 g of potassium iodide and 0·5 g of sodium bicarbonate. Dilute the solution to exactly one litre.

B-5.1.3 *Starch Indicator* — Triturate 5 g of pure starch and 0·01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

B-5.2 Procedure — Take by means of a pipette 2 ml of dilute hydrochloric acid in a porcelain dish. Discharge the sample cooled below 27°C through a cooling coil into a bottle of capacity approximately 250 ml, the end of the tube being placed near the bottom. Allow a quantity of the sample approximately ten times the volume of the bottle to overflow the bottle to ensure a representative specimen of the sample to be obtained. Pour contents of the completely filled bottle into the porcelain basin. Add one millilitre of starch indicator and titrate with constant stirring with standard iodate-iodide solution until a faint blue colour persists in the solution. Measure accurately the capacity of the bottle used.

B-5.3 Calculation

$$\text{Sodium sulphite (as } Na_2SO_3 \text{), parts per million} = 1\,250\,\frac{v}{V}$$

where

v = volume in ml of standard iodate-iodide solution used, and

V = volume in ml of the sample contained in the bottle.

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* Since revised.

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